High pressure component in the APS renewal plan Guoyin Shen, July 25, 2008

Pressure is a principal variable for directing and controlling matter as well as for the synthesis of novel materials. High pressure (HP) has become a major component at synchrotron facilities, because the brilliant synchrotron radiation is an ideal source for small sample volumes in HP environments. At APS, high pressure has been successfully integrated with a number of synchrotron techniques, with significant developments in maximizing brilliance, stability, detecting efficiency, and spatial resolution, minimizing background noise and unwanted signals. The established frontier at many beamlines will be greatly advanced from the overall APS upgrade in x-ray source, beamline optics, advanced detectors, and software controls. In addition, I point out several areas that may be unique to HP and particularly important for advancing to the next generation HP synchrotron research.

Improving depth resolution: While the spatial resolution in a plane perpendicular to x-ray beam has been effectively improved by reducing a probing size, the spatial depth resolution along the x-ray beam remains an under-developed area. Yet, this should be the most important consideration for all HP experiments, because HP samples are always surrounded by chamber materials which often cause strong background signals. Improving depth resolution not only will greatly increase the counting efficiency due to the improvement of S/N ratio, but will make a number of cutting edge projects feasible. The conventional pinhole collimator is often used on the detection side in HP studies, which typically provides a depth resolution of 300-500 μm. The newly developed x-ray lens (e.g., polycapillary x-ray focusing optics) may be used to construct a confocal microscope for collecting scattering signals. Such an x-ray lens can collect a large solid angle (up to 20 degree) with a focus spot as small as 10 μm. The use of x-ray microscope on the detection side will improve the depth resolution by at least an order of magnitude, and will significantly reduce the background from surrounding materials and enhance the detection sensitivity by >100 times. The depth resolution gain through an x-ray microscope is particularly beneficial in HP IXS.

Sub-micron beam size: Since pressure is defined as force per unit area, ultrahigh pressures can be reached by minimizing the area at the tip of diamond anvils. However, this is limited by the minimum available beamsize, which is typically 5-10 μm at HP beamlines around the world. However, beam sizes of a few tens of nm have been realized at several specialized beamlines and the implementation of these sub-micron beams for high-pressure measurements could facilitate x-ray measurements at unprecedented static pressures measured in terapascals. In addition, this rare opportunity for an order-of-magnitude improvement in the spatial resolution of the probe will enable many other new cutting-edge areas, and bring HP synchrotron x-ray diffraction, spectroscopy, and tomography to a new level.

Specialized HP vessels: The diamond anvil cell is almost ubiquitous at HP x-ray facilities around the world. It's success stems from an intrinsically simple conceptual design, yet, for certain specific x-ray techniques, there is a clear demand for a more optimized HP vessel. For instance, for poorly scattering samples, stringent signal to noise limitations will drive the development of a new generation of pressure cells with two primary foci: to minimize the amount of illuminated anvil material and to maximize the sample volume for a given pressure. Innovative pressure cell designs will open up new frontiers in data quality, enabling studies of systems that are currently flux limited, even at 3rd generation synchrotron sources. Significant

effort needs to be spent on developing these optimized HP vessels and integrating these with specialized beamlines and x-ray techniques.

Portable and dedicated systems: In addition to HP vessels, HP experiments often require other ancillary equipment, such as ruby fluorescence system for pressure measurement, laser heating system for heating sample, cryostat for low temperature, and membrane system for precise pressure control. This equipment is often dedicated at HP beamlines. By making them portable, all other beamlines can have access to such equipments for HP studies. The idea is similar to the detector pool at APS. Having a pool of portable systems will boost the HP activities at APS and enable a series of new HP studies with specialized x-ray techniques that are not available at dedicated HP beamlines.

State-of-art central HP facility: Successful HP experiments depend critically upon the quality of sample preparation. Next-generation HP research will require focused effort on micromachining and micro-manipulating techniques in sample preparation. For example, we are now facing grand challenges to fabricate single crystals of various materials to meet stringent requirements of shape, size, and crystal perfection. Techniques such as high pressure single crystal diffraction, inelastic x-ray scattering and charge/spin-density-wave x-ray diffraction, all require perfect single crystals without background scattering from damaged surfaces. This constitutes a unique challenge when the ideal HP sample is typically plates of several microns thickness and several tens of microns width. Femtosecond (FS) laser micromachining plus ion-milling surface cleaning shows promise, since it provides high quality micromachining of many materials and abilities for minimal damage and precise processing. This will also benefit the strain-free cutting of silicon and diamond single crystals for the Optics Fabrication and Metrology Group at APS.